



IN REPLY REFER TO:

United States Department of the Interior

U.S. GEOLOGICAL SURVEY

Box 25046 M.S. 407

Denver Federal Center

Denver, Colorado 80225

NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1997.01

November 5, 1996

To: Chief, Office of Water Quality
Assistant Chief, Office of Water Quality
Assistant Chief Hydrologist for Technical Support
Regional Hydrologists
Acting Chief, NAWQA
Chief, Office of Ground Water
Assistant Chief, Office of Ground Water
Area Hydrologists
District Chiefs
Regional Water Quality Specialists
Assistant Regional Hydrologists for NAWQA
District Water Quality Specialists
Chiefs, NAWQA Study Units
Chief, Quality Water Service Unit, Ocala
Chief, Yucca Mountain Project
QA Manager, Yucca Mountain Project
Chief, Branch of Technical Development & Quality Systems
Employees, National Water Quality Laboratory

From: Peter F. Rogerson, Chief
National Water Quality Laboratory
Branch of Analytical Services

Subject: Use of Syringes to Add Volatile Organic Compounds to Water Samples for Use as Matrix Spike Samples

Authors: Mark W. Sandstrom, Chief, Methods Research & Development Program (SANDSTRO)
(303) 467-8086, and
Bruce R. Darnel, Quality Management Program (BRDARNEL) (303) 467-8089

Revision: None

Supplemental: NWQL Technical Memorandum 97.01S on the Web at: <http://wwwnwql.cr.usgs.gov/>

Commencing with this issue, technical memorandums too lengthy for release via electronic mail have more in-depth versions on the Web. Supplemental tech memos are indicated by a reference line in the heading. The Web version will include an "S" after the number. Paper copies will continue to be mailed upon request.

INTRODUCTION

The National Water Quality Laboratory (NWQL) recommends a new procedure for using a syringe to add Volatile Organic Compounds (VOCs) to matrix spike samples. On-site and laboratory studies comparing the use of syringes and micropipets for spiking water samples with VOCs were conducted by the NWQL and the National Water Quality Assessment (NAWQA) Program. Syringes were found to yield significantly improved recoveries and variabilities for fortifying ground- and reagent-water samples in comparison to the micropipets currently in use. No significant disadvantages were reported or observed with the syringe selected for on-site use.

BACKGROUND

On-site matrix spikes are a type of quality control (QC) sample that provides the information necessary to evaluate biases and interferences from sample matrices as well as biases and variations arising from other processes that may occur from the time the sample vial is preserved on-site until it is analyzed at the laboratory. Data from on-site matrix spikes supplement and complement the laboratory QC samples and provide project- and program-specific QC information but are not used to replace the laboratory QC samples. Comparison of on-site and laboratory QC results might be needed to identify the sources of bias.

Micropipets were previously recommended and provided in NWQL spike kits for VOCs because of concerns regarding difficulty in handling, durability, and potential for cross contamination if syringes were used. The fixed-volume micropipet was thought to be more suitable for use on-site than a syringe for accurately measuring a fixed volume. In addition, the syringe was considered to be less durable because of the potential for bending the plunger and needle. Finally, the syringe needed to be rinsed between samples--a potential source of contamination that could be avoided with the disposable bores of the micropipet.

Despite these advantages, the micropipet provided lower recoveries than the syringe. The reasons for this difference are not well understood, although the recoveries using the micropipet decrease as compound volatility increases. This process introduces an additional source of bias for interpreting the effects of matrix or bias from shipping, unless the results are compared to laboratory QC samples prepared using the micropipet. This comparison creates extra work for the laboratory because the laboratory QC samples are routinely prepared with a syringe. While not ideal, the micropipet can provide information regarding bias from matrix or shipping when compared to reagent water samples prepared using the micropipet. Recent experiments were conducted to determine if a robust syringe procedure could be developed for use on-site that could overcome some of the difficulties mentioned previously.

These experiments indicated that bias and variability could be minimized by using a Hamilton 1810RN syringe. The mean relative standard deviation (RSD) of VOC recoveries was 15 percent for the micropipet and 8 percent for the syringe. The overall mean recovery of VOCs using the micropipet was 70 percent, and the overall mean recovery using the syringe was 90 percent.

Analytical results of the on-site and laboratory comparison of syringe and micropipet used to add VOCs to ground- and reagent-water are available as supplementary information (NWQL Technical Memorandum 97.01S) on the NWQL WWW site (URL: <http://wwwnwql.cr.usgs.gov/USGS>). The performance of the micropipet used with reagent water samples is also summarized. Those data provide reference information that can be used to interpret VOC matrix spike samples previously prepared with the micropipet.

SUMMARY

Syringes were found to yield significantly improved recoveries and variabilities for fortifying ground- and reagent-water samples in comparison to the micropipets currently in use. The process of spiking VOCs with a micropipet is a significant source of bias. The average recovery and reproducibility of VOCs spiked with a syringe in the laboratory and on-site were significantly improved in comparison to the micropipet. No significant disadvantages were reported or observed with the use of the syringe selected for evaluation on-site.

Note: The new procedure using a syringe is recommended **ONLY** for VOCs; micropipets will continue to be used for spiking pesticides and other organic compounds.

Syringes or spike kits with syringes can be obtained from the NWQL by E-mail request to DENSUPPL.

Impact on Data Base: None

Supersedes: None

Supplementary Info: NWQL Tech Memo 97.01S on the Web (see address below)

Key words: field spike, micropipet, VOC, syringe, QA/QC sample

Distribution: See above plus the Netnews usgs.labnews & .water.quality, WRD Secretaries; Field and Project Offices; Hydrologic Technicians; and <http://wwwnwql.cr.usgs.gov/USGS>